Chapter

11

Statistical Optimization

Large number of experimental run is required to develop the input-output relationship of any process. Utilization of statistical methods can reduce the number of the experimental run. This chapter elucidates the design of experiment for optimize use of experimental data. Response surface methodology is used to optimize the process output. A process plant can be optimized efficiently using these methods.

11.1 Design of Experiment

A huge number of experiments are required in research, development and optimization of any system. This research is carried out in labs, pilot plants or full-scale plants. An experiment may be a model based experiment or experiment with the physical system [Lazić, (2004)]. When the experimentation cost is very high, it is very difficult for the researcher to examine the numerous factors that have an effect on the processes using trial and error methods within a short time and limited resources. As an alternative, we need a technique that identifies some key factors in the most effective way. Then this leads the process to the best setting to satisfy the increasing demand for increased productivity with improved quality.

A one-factor-at-a-time experiment is used to study the effect of various factors during experimentation. This method involves changing a single factor at a time to analyze the impact of that factor on the product or process output. The main advantage of "one-factor-at-a-time" experiment is that we can perform the experiment easily. Despite this fact, these methods do not permit us to investigate of how a factor influences the process or a product in the presence of other factors. When the response of a process is changed due to the existence of one or more other factors, that relationship is called an interaction. Sometimes the effects of interaction terms are more significant compared to the individual effects. Because in the application environment of the process or product, most of the factors present together rather than the isolated incidents of single factors at different times. A chemical reacting system can be considered as an example of interaction between two factors. The reaction rate slightly increases when the temperature is increased and

there is no effect of pressure on reaction rate. However, the reaction rate changes rapidly when both the temperature and pressure are changing simultaneously. In this system, an interaction does exist between the two factors that affect the chemical reaction. The major disadvantage of the one-factor-at-a-time strategy is that it is not able to consider any probable interaction between various factors. An interaction is the failure of the one factor to provide the same influence on the response at various levels of another factor [Montgomery (2001)]. The DOE technique guarantees that all factors and their interactions are investigated methodically. The one-factor-at-a-time experiments overlook the interactions and may direct to misleading outcomes. Therefore, information collected from the analysis of DOE is much more complete and reliable.

As the experiment is considered an essential part of science and technology, the question of efficiency of utilizing an experimental data is therefore, necessary. It is found that very less percentage of experimental data is used efficiently. To enhance the efficiency of research, it is essential to introduce something novel into conventional experimental research. The degree of precision and completeness of experimental data and information about the system that is being tested are used to determine the efficiency of experimental research. Fisher [Fisher 1926,1958,1960] methodically introduced the idea and theories of statistics into designing experimental investigations, which includes the concept of factorial design and the analysis of variance.

Even though applications of statistical design started in1930s, it was accelerated by the development of response surface methodology (RSM) by Box and Wilson (1951).

For the design of experiment, the main purposes are:

i. Decrease or minimization of total number of experiments
ii. Varying of all factors simultaneously that formalizes the activities of experimenter
iii. Selection of a proper strategy that facilitates reliable solutions to be found after each series of experiments

Therefore, the following factors should be considered before a design can be chosen

a. Choice of factor, levels, and range
b. Selection of the response variable
c. Selection of experimental design
d. Performing the experiment efficiently
e. Statistical analysis of the data

11.1.1 Stages of DOE

The DOE method consist five steps namely planning, screening, optimization, robustness testing and verification.

i. Planning

Before starting the process of data collection and testing, it is essential to plan the experimentation very carefully. The usefulness of any experimental data depends on how it is designed. During planning, we have to keep in mind some of these considerations i) a thorough and precise objective identifying is required to perform the investigation, ii) estimation of resources and time available to accomplish the objective and iii) collection of prior experience with the experimentation method.

We have to create a team that will identify probable factors for investigation and the most suitable response(s) to measure. This team should be comprised of individuals from various disciplines related to the process or product. The knowledge from various disciplines can be gathered from those team members. The teamwork strategy encourages synergy, which provides a richer set of factors for analyzing and consequently a more comprehensive experiment. We will get better understanding of the process or product when the experiment is planned carefully. During implementation, a well-planned experiment is easy to perform and evaluate. Conversely, botched experiments may result in inconclusive data sets that may not be possible to analyze even with an excellent statistical tool (www.weibull.com).

ii. Screening

The DOE method starts with the screening of variables. Screening experiments are required to distinguish the significant factors that influence the process under consideration out of the large pool of potential factors. Prior knowledge of the process is required to perform the experiments that eliminate insignificant factors and focus interest on the key factors that require further thorough analyses. This procedure use a fractional factorial design, since only a fraction of the possible values of the corners are investigated. Very few executions are required as screening experiments are usually efficient designs. Our focus is not on studying the effect of interactions but on identifying the vital few factors.

iii. Optimization

After proper selection of the relevant factors that are influencing the process, the next step is to decide the best setting of these factors to produce the preferred objective. The objective of the study depends on the process or product under investigation, it may be either to increase yield or to decrease variability or to get settings that will give us both at the same time.

iv. Robustness Testing

Robustness is defined as the degree to which a system operates correctly in the presence of exceptional inputs or stressful environmental conditions [IEEE Std 24765: 2010]. After deciding the optimal settings of the factors, it is required to make the process or product insensitive to variations that may arise in the application environment. These variations occur due to the fluctuations in factors that influence the process, but they are beyond our control. These factors (e.g., humidity in atmosphere, ambient temperature, variation in raw material, etc.) are called the uncontrollable factors or noise. It is necessary to recognize the sources of these variations and take actions to assure that the process or product is made insensitive (or robust) to these factors.

v. Verification

Validation of the best settings is required to verify that whether the process functions as desired and all objectives are satisfied. The final stage involves this validation by performing a small number of follow-up experimental runs.

11.1.2 Principle of DOE

The three basic stages of experimental design are replication, randomization, and blocking. The basic experiment is repeated by using replication. There are two essential properties of replication. The first one is that it helps the experimenter to get an assessment of the experimental error. Secondly, whenever the sample mean ($\bar{y}$) is employed to evaluate the effect of a factor in the

experiment, replication allows the experimenter to get a more accurate estimation of this effect. For example; if σ^2 is the variance of an individual observation and n is the number of replicates, the variance of the sample mean is

$$\sigma_{\bar{y}}^2 = \frac{\sigma^2}{n} \tag{11.1}$$

Randomization is the fundamental issue for using any statistical methods in experimental design. Using randomization process, we randomly determine both the distribution of the experimental material and the order in which the individual runs or trials of the experiment are to be executed. In statistical methods, the observations (or errors) should be independently distributed random variables. By randomization, we make this assumption valid. We also help in "average out" the effects of extraneous factors that may be present by properly randomizing the experiment [Montgomery, (2001)].

Blocking is a procedure that is employed to increase the accuracy with which comparisons are made among the factors of interest. Blocking is also used to diminish or remove the variability contributed by nuisance factors; that the factors may affect the experimental response but in which we are not interested directly. For instance, to complete all experimental runs in a chemical process industry it may require more than one batch of raw material. Though, there might be variations between those batches due to source to source variability. Whenever we are not interested to study particularly this effect, we can consider these differences between the batches of raw material as a nuisance factor. Usually, a set of relatively homogeneous experimental conditions is considered as a block. The variability within the same batch of raw material would be expected smaller than the variability between different batches. Therefore, each batch of raw material would form a block in the chemical process industries.

Computer software programs are extensively used for assisting experimenters to select and construct experimental designs. Commercial software like MINITAB (chapter 12) is available to design the experiment.

Example 11.1

A chemical engineer is interested to investigate the reaction rate as a function of pH.

This is a single factor experiment with 5 level of the factor and 5 replicates. We conducted the experiment with five different pH level and repeated 5 times in each pH level.

Table 11.1 Single factor experiment with 5 level of the factor and 5 replicates

pH	Experimental run number				
3	1	2	3	4	5
5	6	7	8	9	10
7	11	12	13	14	15
9	16	17	18	19	20
11	21	22	23	24	25

Now we arrange the experimental number randomly (randomization). After randomization, the 5th observation comes first. The randomized data is given in Table 11.2.

Table 11.2 Arrangement of experimental run after randomization

Test sequence	Run number	pH
1	5	3
2	7	5
3	21	11
4	16	9
5	3	3
6	19	9
7	20	9
8	25	11
9	2	3
10	15	7
11	4	3
12	9	5
13	18	9
14	10	5
15	14	7
16	1	3
17	17	9
18	6	5
19	23	11
20	12	7
21	8	5
22	13	7
23	22	11
24	11	7
25	24	11

the data from reaction rate (mol × 10^{-2}/L.s) is given below

Table 11.3 Reaction rate data at different pH

pH	Observations (Reaction rate, mol×10^{-2}/L.s)						
	1	2	3	4	5	Total	Average
3	2	3	2	5	3	15	3
5	3	4	3	5	4	19	3.8

7	5	5	4	6	5	26	5.2
9	4	3	3	4	4	18	3.6
11	2	3	2	3	3	13	2.6
						91	3.64

11.1.3 ANOVA study

"Analysis of Variance" (ANOVA) is a technique that employs tests based on variance ratios of several groups of observations. In this analysis, it is decided if significant differences are there among the means of these groups of observations wherein each group follows a normal distribution. Whenever three or more means are there, the analysis of variance method extends the *t*-test applied to determine whether there is a difference between two means.

Decomposition of the Total Sum of Squares

Consider, we have '*a*' treatments or different levels of a single factor that we would like to compare. The observed response from each of the '*a*' treatments is a random variable. The data can be represented as in Table 11.3. This name Analysis of Variance is obtained from a partitioning of the total variability into its component parts. The total corrected sum of squares can be written as

$$SS_T = \sum_{i=1}^{a}\sum_{j=1}^{n}\left(y_{ij} - \overline{y}\right)^2 \tag{11.2}$$

the overall variability in the data can be measured by using Eq. (11.2).

The total corrected sum of squares SS_T can be represented as

$$\sum_{i=1}^{a}\sum_{j=1}^{n}\left(y_{ij} - \overline{y}\right)^2 = \sum_{i=1}^{a}\sum_{j=1}^{n}\left[\left(\overline{y}_i - \overline{y}\right) + \left(y_{ij} - \overline{y}_i\right)\right]^2 \tag{11.3}$$

or

$$\sum_{i=1}^{a}\sum_{j=1}^{n}\left(y_{ij} - \overline{y}\right)^2 = n\sum_{i=1}^{a}\left(\overline{y}_i - \overline{y}\right)^2 + \sum_{i=1}^{a}\sum_{j=1}^{n}\left(y_{ij} - \overline{y}_i\right)^2 + 2\sum_{i=1}^{a}\sum_{j=1}^{n}\left(\overline{y}_i - \overline{y}\right)\left(y_{ij} - \overline{y}_i\right) \tag{11.4}$$

In Eq. (11.4), the cross-product term is zero, because

$$\sum_{j=1}^{n}\left(y_{ij} - \overline{y}_i\right) = y_i - n\overline{y}_i = y_i - n\left(y_i/n\right) = 0 \tag{11.5}$$

Therefore, we have

$$\sum_{i=1}^{a}\sum_{j=1}^{n}\left(y_{ij} - \overline{y}\right)^2 = n\sum_{i=1}^{a}\left(\overline{y}_i - \overline{y}\right)^2 + \sum_{i=1}^{a}\sum_{j=1}^{n}\left(y_{ij} - \overline{y}_i\right)^2 \tag{11.6}$$

The total variability in the data as shown in the Eq. (11.6), is measured by the total corrected sum of squares, can be splitted into a sum of squares of the differences between the treatment averages and the grand average (1st term on the right side of Eq. (11.6)), plus a sum of squares of the differences of observations within treatments from the treatment average (2nd term on the right side of Eq. (11.6)). Now, the differences between treatment means represent the difference between the observed treatment averages and the grand average, while the differences of observations within a treatment from the treatment average can be due only to random error. Therefore, we can write Eq. (11.6) as

$$SS_T = SS_{\text{Treatments}} + SS_E \tag{11.7}$$

Example 11.2

To discuss the analysis of variance, we will consider the reaction rate data given in Table 11.3.

Calculate the value of SS_T from Eq. (11.2)

$$SS_T = \sum_{i=1}^{a}\sum_{j=1}^{n}\left(y_{ij} - \bar{y}\right)^2 \tag{11.2}$$

$$SS_T = \sum_{i=1}^{5}\sum_{j=1}^{5} y_{ij}^2 - \frac{y^2}{N} \tag{11.8}$$

$$= (2)^2 + (3)^2 + \cdots + (3)^2 + (3)^2 - \frac{(91)^2}{25}$$

$$= 354 - 331.24 = 22.76$$

and calculate the value of $SS_{\text{Treaments}}$ from Eq. (11.9)

$$SS_{\text{Treaments}} = \frac{1}{n}\sum_{i=1}^{5} y_i^2 - \frac{y^2}{N} \tag{11.9}$$

$$= \frac{1}{5}\left[(15)^2 + (19)^2 + (26)^2 + (18)^2 + (13)^2\right] - \frac{(91)^2}{25}$$

$$= 351 - 331.24 = 19.76$$

from Eq. (11.7), we get

$$SS_E = SS_T - SS_{\text{Treatments}} \tag{11.10}$$

$$SS_E = 22.76 - 19.76 = 3.0$$

Where, $SS_{\text{Treaments}}$ is called the sum of squares due to treatments and SS_E is called the sum of squares due to error.

The value of F_0 is calculated using the Eq. (11.11)

$$F_0 = \frac{SS_{\text{Treatments}}/(a-1)}{SS_E/(N-a)} = \frac{MS_{\text{Treatments}}}{MS_E} \tag{11.11}$$

$$= 32.933$$

11.1.4 Types of experimental design

The experimental designs are classified based on different characteristic like geometric configuration, level of experiments, etc.

11.1.4.1 First-order designs

The most familiar first-order designs are 2^k factorial (k is the number of control variables), Plackett–Burman, and simplex designs.

Factorial Designs

A factorial design is a type of designed experiment that allows us to study the effects that various factors can have on a response. A factorial experiment is an experimental approach wherein the design variables are varied simultaneously, rather than one at a time. When we are conducting an experiment, varying the levels of all factors at the same time rather than one at a time that allows us to study the interactions between the factors. The factorial designs are broadly classified into two groups namely full factorial design and fractional factorial design.

Full Factorial Designs

A full factorial design is a design strategy, where researchers measure responses at all combinations of the factor levels. Figure 11.1 represents a two level design of three variables (X_1, X_2, and X_3). The number of available combinations is eight.

Fractional Factorial Designs

A fractional design is a design strategy in which experiments are conducted only for a selected subset or "fraction" of the runs in the full factorial design. When resources are limited or the number of factors in the design is large, the fractional factorial designs are a good choice because

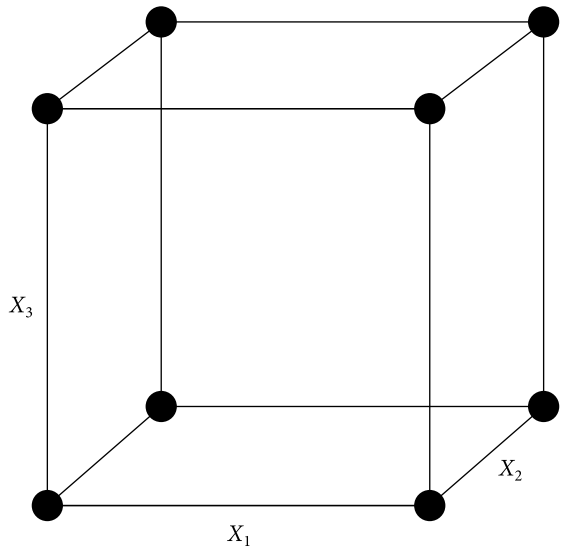

Fig. 11.1 Full factorial design with three variables

they use fewer runs than the full factorial designs. As fractional factorial design uses a subset of a full factorial design, some of the main effects and 2-way interactions are confounded and cannot be partitioned from the other higher-order interaction effects. Usually experimenters are assuming that the higher-order effects are negligible in order to get information about major effects and considering the lower-order interactions with fewer runs. In Fig. 11.2, a ½ fractional factorial design with 3 factors and 2 levels has been shown. Only four points are considered as design points.

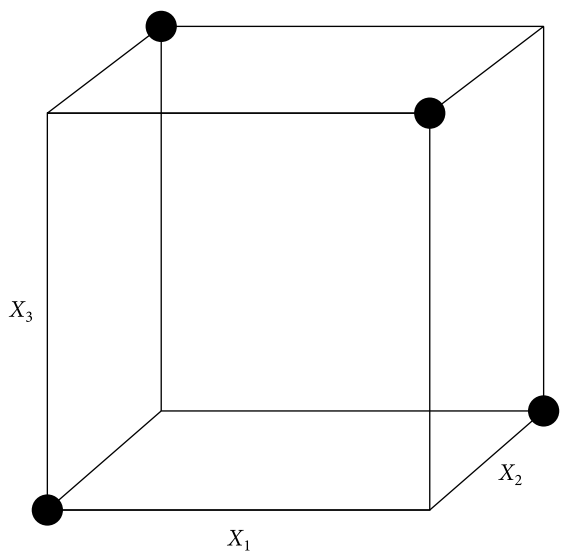

Fig. 11.2 Fractional factorial design with three variables

Plackett-Burman Designs

The Plackett-Burman design is a standard two-level design as active screening design, which offers the option of 4, 8, 16, 32 or more trials-runs, but only the power of two. When the number of variables is large and the aim is to choose the most significant variables for further experimentation, usually only the main effects are of interest. In such cases, the most cost effective alternative is to use the designs that have as many experiments as there are parameters. This is a popular and economical approach that gives information only on the effects of single factors, but not on interactions. The Plackett–Burman designs (1946) are two level fractional factorial designs for studying $k = N - 1$ variables in N number of runs, where N is a multiple of 4. For $N = 12$, 20, 24, 28 and 36, sometimes the Plackett–Burman designs are significant, as they fill gaps in the standard designs. Unfortunately, the structure of these particular Plackett–Burman designs are very messy alias. For instance, the very popular 11th factor in the 12-runs choice causes each main effect to be partially aliased with 45 two-factor interactions. Theoretically, we can escape from this if absolutely there exist no interactions, although this assumption is a very dangerous. The unexpected aliasing takes place with many Plackett–Burman designs. Therefore, it is suggested to keep away from them in favor of the standard two-level designs [Lazic, (2004)].

Since these designs cannot be represented as cubes, sometimes they are called non-geometric design. Some of the software packages (e.g., MINITAB) for design of experiments are available with Plackett–Burman designs.

11.1.4.2 Second-order designs

The most popular second-order designs are Central Composite Design and Box-Behnken Design. Following section discusses these methods.

Central composite design

The Central Composite Design (CCD) has been extensively utilized as the experimental design. For fitting a quadratic surface, the CCD is a very efficient. It helps us to optimize the effective parameters with a minimum number of experiments, and also to analyze the interaction between the parameters. The CCD consists of a 2^k factorial runs with $2k$ axial runs and n_0 center runs. In CCD, each variable is investigated at two levels and as the number of factors, k, increases the number of runs for a complete replicate of the design increases rapidly.

The total number of experiment require is given by

$$N = 2^k + 2k + n_0 \tag{11.12}$$

The different level for each experiment is $-\alpha,\ -1,\ 0,\ +1,$ and $+\alpha$

where

$$\alpha = 2^{k/4} \tag{11.13}$$

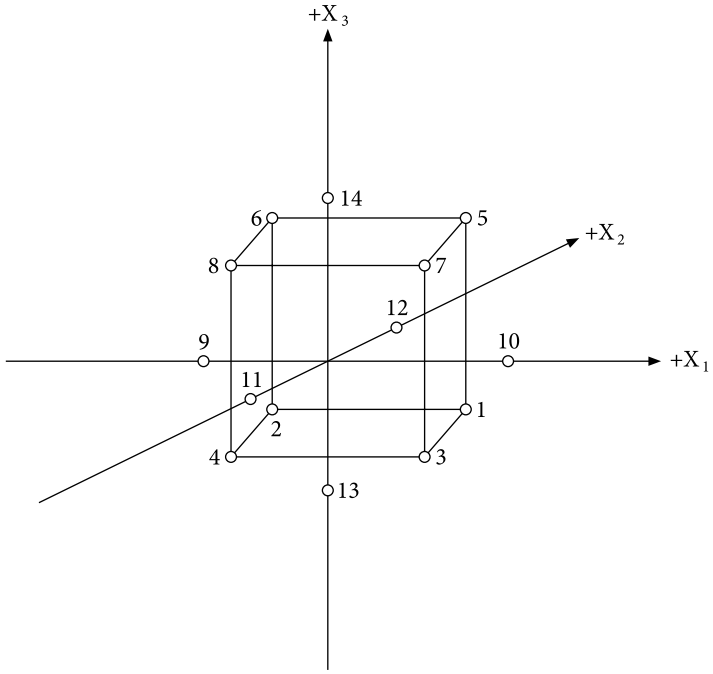

Fig. 11.3 Central composite design

Rotatability

It is important for the second order model to provide good predictions throughout the region of interest. One way to define "good" is require that at points of interest **x**, the model comprise a reasonably consistent and stable variance of the predicted response. The variance of the predicted response at some point x is

$$V\left[\hat{y}(\mathbf{x})\right] = \sigma^2 \mathbf{x}'\left(\mathbf{X}'\mathbf{X}\right)^{-1}\mathbf{x} \tag{11.14}$$

Box and Hunter (1957) suggested that a second order response surface design should be rotatable. This means that the $V\left[\hat{y}(\mathbf{x})\right]$ is the same at all points **x** that are the same distance from the centre of the design. That is, the variance of predicted response is constant on spheres. A design with this property will leave the variance of $\hat{y}$ unaffected when we rotate the design about the centre (0, 0,…,0), hence, the name rotatable design. Rotatability is a reasonable basis for the selection of a response surface design. Because RSM is used for optimization and the position of the optimum point is unknown to us before conducting the experiment, it makes sense to use a design that gives equal precision of estimation in all directions (it can be shown that any first order orthogonal design is rotatable). A central composite design is made rotatable with proper selection of α. The value of α for rotatability depends on the number of points in the factorial portion of the design; actually, $\alpha = \left(n_F\right)^{1/4}$ give us a rotatable central composite design where n_F is the number of points used in the factorial portion of the design.

Box–Behnken design

Box and Behnken (1960) have proposed some three-level designs for fitting response surfaces. The main advantage of Box–Behnken design over CCD is that it requires less number of experimental design points which reduces the experimental cost, consequently cost of optimization process. Figure 11.4 shows the geometric representation of Box–Behnken (B–B) design. This design is a spherical in shape where all points are lying on a sphere of radius $\sqrt{2}$. The B–B design does not have any points at the vertices of the cubic region constructed by the lower and upper limits for each variable. Sometimes this design could be helpful particularly when the points on the corners of the cube represent factor-level combinations that are extremely expensive or sometimes impossible to test owing to physical process constraints.

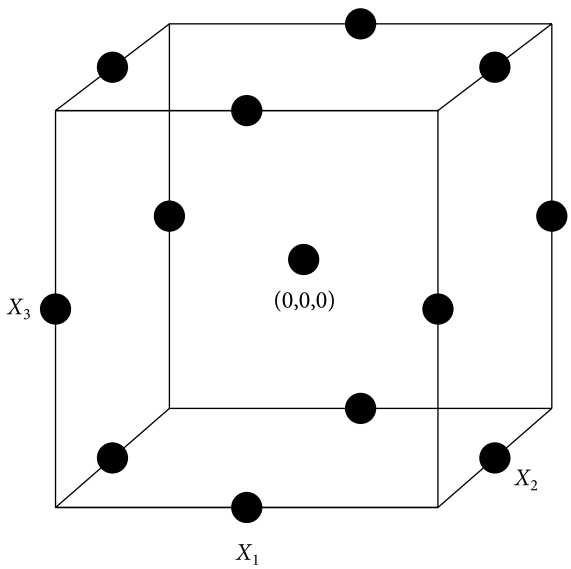

Fig. 11.4 Box–Behnken design

Table 11.4 Three variable B–B design

Run	x_1	x_2	x_3
1	–1	–1	0
2	1	–1	0
3	–1	1	0
4	1	1	0
5	–1	0	–1
6	1	0	–1
7	–1	0	1
8	1	0	1

9	0	−1	−1
10	0	1	−1
11	0	−1	1
12	0	1	1
13	0	0	0
14	0	0	0
15	0	0	0

The value in table 11.4 is in coded form, which is given by the following equation

$$\text{Coded value} = X_i = \frac{\left(x_i - \overline{x}_i\right)}{\Delta x} \tag{11.15}$$

where Δx is called step change.

11.1.4.3 D-optimal design

There are many popular design optimality criteria; probably the most widely used is the D-optimality criterion. A design is called D-optimal if $\left|\left(\mathbf{X}'\mathbf{X}\right)^{-1}\right|$ is minimized. It turns out that a D-optimal design minimizes the volume of the joint confidence region on the vector of regression coefficients. A measure of the relative efficiency of design 1 to design 2 according to the D-criterion is represented by

$$D_e = \left(\frac{\left|\left(\mathbf{X}_2'\mathbf{X}_2\right)^{-1}\right|}{\left|\left(\mathbf{X}_1'\mathbf{X}_1\right)^{-1}\right|}\right)^{\frac{1}{p}} \tag{11.16}$$

where $\mathbf{X}_1$ and $\mathbf{X}_2$ are the $\mathbf{X}$ matrices for the two designs and p is the number of model parameters.

11.2 Response Surface Methodology

Response surface methodology (RSM) is a compilation of mathematical and statistical techniques. RSM is a method for designing experiments, building models, evaluating the effects of various factors and reaching the optimum conditions with a limited number of planned experiments [Khuri and Cornell (1996)]. By designing the experiments carefully, our aim is to optimize a response (output variable) which is influenced by various independent variables (input variables). RSM shows how a given set of input variables affect a particular response within the specified region of interest, and what values of input variables will yield an optimizer for a particular response. Initially RSM was developed to find out optimum operating conditions in the chemical process industry, but now it is employed in a variety of fields and applications, not only in the engineering and physical sciences, but also in clinical, social, and biological sciences [Khuri (2001)]. The main aim of RSM

is the optimization of process. This could imply, for instance, the operating cost minimization of a production process, the minimization of the variability of a quality characteristic, maximization of the yield of a chemical process, or achieving the desired specifications for a response. It is frequently considered the multiple responses of interest in practical problems in chemical industry [Castillo, (2007)]. The main advantage of RSM is that, it formulate the optimization problem with very less cost of analysis methods and their associated numerical noise. For optimization using RSM, the first step is design the experiment and develop the response surface based on experimental data. Design of experiment is discussed in the previous section (11.2).

For optimizing an industrial process, RSM techniques suggest to construct a parametric model for the expected response using designed experiments. Different model can be considered to find the relationship between response and variables. The response function can be represented as

$$y = f\left(x_1, x_2, x_3\right) + \varepsilon \tag{11.17}$$

where ε is the error or noise observed in the response y. If the expected response is represented by $E\left(y\right) = f\left(x_1, x_2, x_3\right) = \eta$, then the surface represented by

$$\eta = f\left(x_1, x_2, x_3\right) \tag{11.18}$$

The linear model is

$$y = \beta_0 + \sum_{i=1}^{k} \beta_i x_i + \varepsilon \tag{11.19}$$

and second order model is

$$y = \beta_0 + \sum_{i=1}^{k} \beta_i x_i + \sum_{i} \sum_{j} \beta_{ij} x_i x_j + \sum_{i=1}^{k} \beta_{ii} x_i^2 + \varepsilon \tag{11.20}$$

Usually the response surface is presented graphically as shown in Figs 11.5a, 11.6a, and 11.7a. We often plot the contours of the response surface for better visualization of the shape of response surface. Contours, also helps us to determine the optimum point.

Example 11.3

Anupam *et al.* [Anupam *et al.* (2011)] have discussed Cr(VI) from water using activated carbon depends on three factors pH, powdered activated carbon dose (PAC) and time of adsorption. They found the correlation for chromium removal as

$$\mathrm{Cr(VI)\ removal}(\%) = f\left(\mathrm{pH, PAC, time}\right)$$

or

$$\begin{aligned}\text{Cr(VI) removal}(\%) &= 77.92 - 8.46(\text{pH}) + 9.41(\text{PAC}) + 3.86(\text{time}) \\ &+0.68(\text{pH})^2 - 3.53(\text{PAC})^2 - 7.33(\text{time})^2 \\ &-1.08(\text{pH})(\text{PAC}) + 1.08(\text{pH})(\text{time}) - 0.452(\text{time})(\text{PAC})\end{aligned} \tag{11.21}$$

Three surfaces can be constructed as shown in Figs 11.5a, 11.6a, and 11.7a. The corresponding contours can be represented by Fig. 11.5b, 11.6b, and 11.7b.

$$\text{Cr(VI) removal}(\%) = f(\text{PAC,time}) \text{ at pH = 0.} \tag{11.22a}$$

$$\text{Cr(VI) removal}(\%) = f(\text{pH,time}) \text{ at PAC = 0} \tag{11.22b}$$

$$\text{Cr(VI) removal}(\%) = f(\text{pH,PAC}) \text{ at time = 0} \tag{11.22c}$$

These surfaces are called response surfaces.

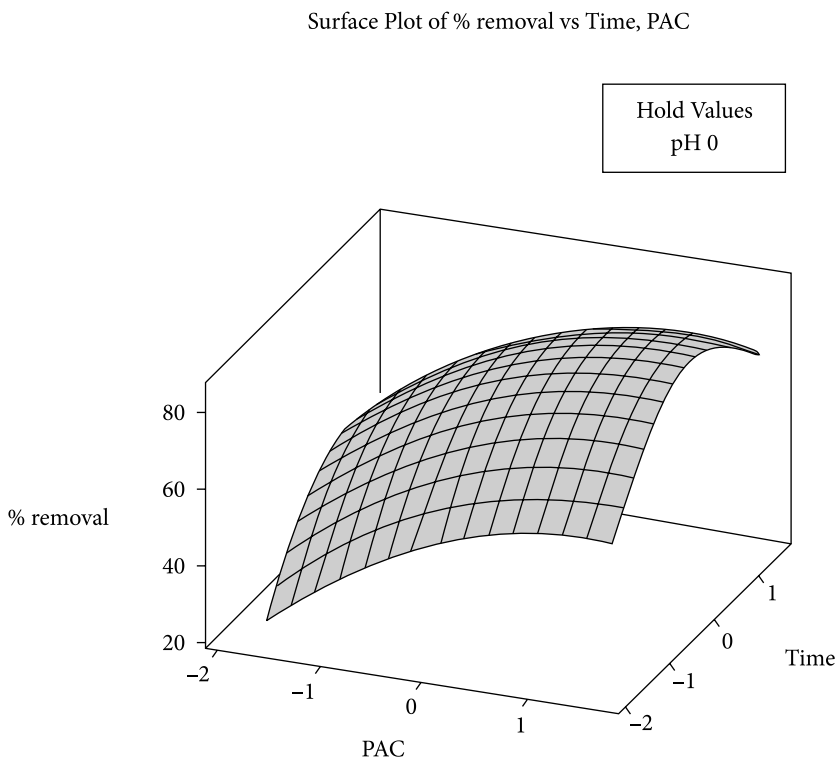

Fig. 11.5a Response surface formed from Eq. (11.22a)

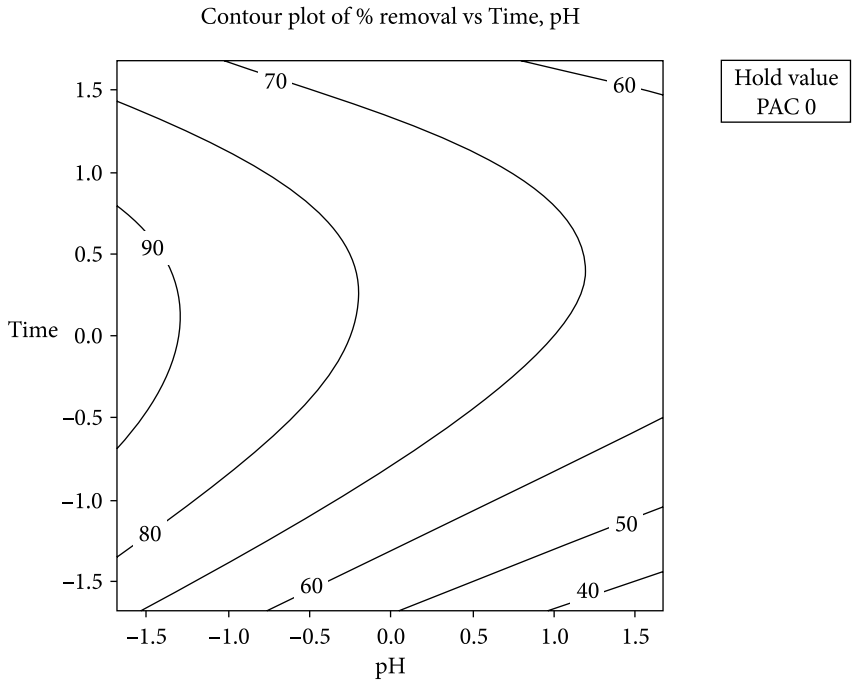

Fig. 11.5b Contour representation of Fig. 11.5a

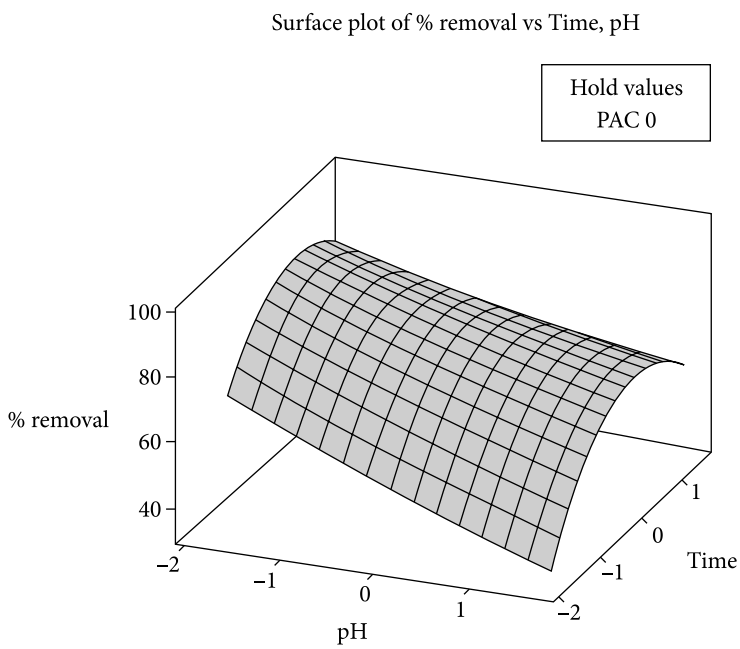

Fig. 11.6a Response surface formed from Eq. (11.22b)

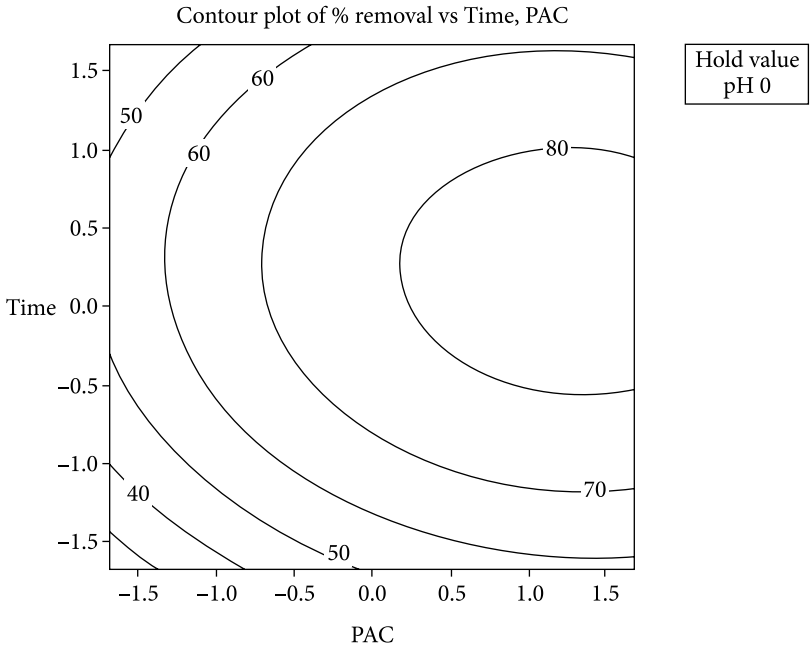

Fig. 11.6b Contour representation of Fig. 11.6a

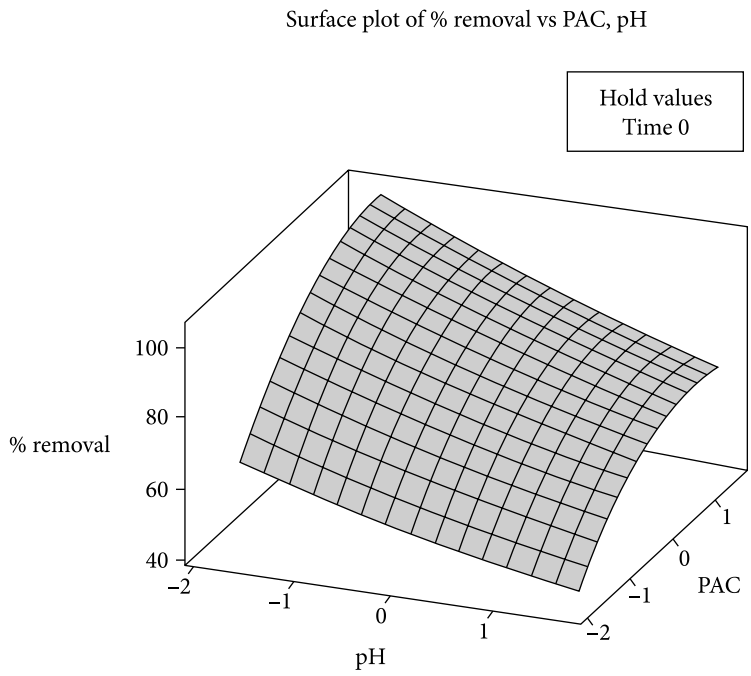

Fig. 11.7a Response surface formed from Eq. (11.22c)

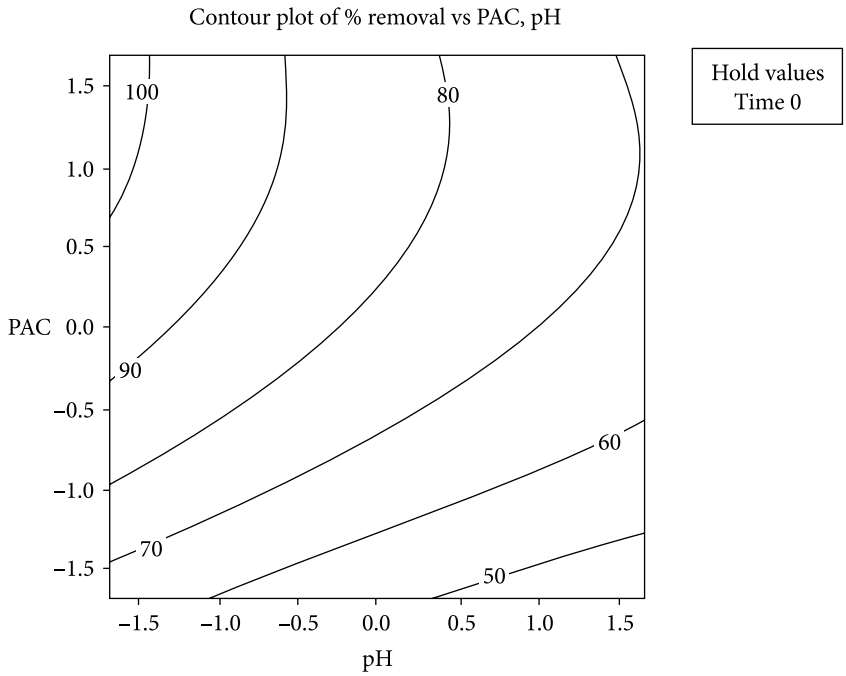

Fig. 11.7b Contour representation of Fig. 11.7a

For a conventional DOE when it is expected that many of the design variables initially considered have small or no effect on the response, screening experiments are performed in the early stages of the process. The purpose is to recognize the design variables that have large effects and need further investigation. Genetic Programming can be used efficiently for screening, genetic algorithm has been demonstrated in Section 9.1.

11.2.1 Analysis of a second order response surface

Suppose we are interested to find the location of the stationary point. We may obtain a general mathematical solution for the same. Writing the second order model in matrix form, we have

$$\hat{y} = \hat{\beta}_0 + \mathbf{x}'\mathbf{b} + \mathbf{x}'\mathbf{B}\mathbf{x} \tag{11.23}$$

where

$$\mathbf{x} = \begin{bmatrix} x_1 \\ x_2 \\ \vdots \\ x_k \end{bmatrix}, \mathbf{b} = \begin{bmatrix} \hat{\beta}_1 \\ \hat{\beta}_2 \\ \vdots \\ \hat{\beta}_k \end{bmatrix} \quad \text{and} \quad \mathbf{B} = \begin{bmatrix} \hat{\beta}_{11} & \hat{\beta}_{12}/2 & \cdots & \hat{\beta}_{1k}/2 \\ & \hat{\beta}_{22} & \cdots & \hat{\beta}_{2k}/2 \\ & & \ddots & \vdots \\ \text{sym} & & & \hat{\beta}_{kk} \end{bmatrix}$$

Here, **b** is a ($k \times 1$) vector of the first order regression coefficients and **B** is a ($k \times k$) symmetric matrix whose main diagonal elements are the pure quadratic coefficients $\left(\hat{\beta}_{ii}\right)$ and whose off-diagonal elements are one-half the mixed quadratic coefficients $\left(\hat{\beta}_{ij}, i \neq j\right)$. The derivative of $\hat{y}$ with respect to the elements of the vector x equated to 0.

$$\frac{\partial \hat{y}}{\partial \mathbf{x}} = \mathbf{b} + 2\mathbf{B}\mathbf{x} = 0 \tag{11.24}$$

The stationary point is the solution of this equation.

$$\mathbf{x}_s = -\frac{1}{2}\mathbf{B}^{-1}\mathbf{b} \tag{11.25}$$

Furthermore, by substituting Eq. (11.25) into Eq. (11.23), we can establish the predicted response at the stationary point as

$$\hat{y}_s = \hat{\beta}_0 + \frac{1}{2}\mathbf{x}_s'\mathbf{b} \tag{11.26}$$

Example 11.4

Find the stationary point for the problem formulated by Anupam *et al.*

Table 11.5 Values of the coefficient (Anupam *et al.*)

Term	Coefficients
Constant	77.92
pH	–8.46
PAC	9.41
time	3.86
pH × pH	0.68
PAC × PAC	–3.53
time × time	–7.33
pH × PAC	–1.08
pH × time	1.88
PAC × time	–0.45

$$\mathbf{b} = \begin{bmatrix} -8.46 \\ 9.41 \\ 3.86 \end{bmatrix} \quad \mathbf{B} = \begin{bmatrix} 0.68 & -0.54 & 0.94 \\ -0.54 & -3.53 & -0.225 \\ 0.94 & -0.225 & -7.33 \end{bmatrix}$$

$$\mathbf{B}^{-1} = \begin{bmatrix} 1.121 & -0.181 & 0.149 \\ -0.181 & -0.255 & -0.015 \\ 0.149 & -0.015 & -0.117 \end{bmatrix}$$

which gives the stationary point (using Eq. (11.25))

$$\mathbf{x}_s = \begin{bmatrix} -5.303 \\ -0.462 \\ -0.929 \end{bmatrix}$$

Characterizing the response surface

Characterization of response surface in the vicinity of this stationary point is necessary. By characterize, we mean determine whether the stationary point is a point of minimum or maximum response or saddle point. We also interested to study the relative sensitivity of the response to the variables $x_1, x_2,\ldots,x_k$.

The most straightforward way to do this is to examine a contour plot of the fitted model. If there are two or three process variables, the construction and interpretation of this contour plot is quite simple. However, even when there are relatively few variables, a more formal analysis, called the canonical analysis, can be useful.

11.2.2 Optimization of multiple response processes

Most of the processes in real life required the optimization with respect to various criteria simultaneously. Sometimes the operating conditions required to satisfy various conditions or constraints on m responses, $y_1,\ldots,y_m$. For example, simultaneous optimization of selectivity as well as production rate for a reacting system.

Desirability Approach

The desirability approach was initially recommended by Harrington [Harrington, (1965)] and later modified by Derringer and Suich [Derringer and Suich, (1980)] to its most familiar form that is in practical use nowadays. The desirability function approach is a well-accepted method in industry for dealing with the multiple response processes optimization. The quality of a process or product that has multiple quality characteristics, with one of them out of some desired limits and that is totally unacceptable. This technique finds the operating conditions x that offer the "most desirable" values of the response [Castillo, (2007)].

For each response $y_i(x)$, a desirability function $d_i(y_i)$ assigns numbers between 0 and 1 to the probable values of y_i. The value $d_i(y_i) = 0$ represents a absolutely undesirable value of y_i and $d_i(y_i) = 1$ represents a completely desirable or ideal response value. To obtain the overall desirability D, the individual desirabilities are combined using the geometric mean as Eq. (11.27):

$$D = \left[d_1(y_1) \times d_2(y_2) \times \ldots \times d_m(y_m) \right]^{1/m} \tag{11.27}$$

where the number of responses is represented by m. It is obvious that the overall desirability is zero whenever any of these responses i is completely undesirable ($d_i(y_i) = 0$). In practical application of this method, the fitted response models $\hat{y}_i$ are used.

Various desirability functions $d_i(y_i)$ can be used depending on whether a particular response y_i is to be minimized, maximized, or assigned to a target value. Derringer and Suich [Derringer and Suich, (1980)] proposed a useful class of desirability functions. Suppose L_i, U_i and T_i be the lower, upper, and target values desired for response i, where $L_i \le T_i \le U_i$. If a response is of the "target is best" type, then its individual desirability function is

$$d_i(\hat{y}_i) = \begin{cases} 0 & \text{if } \hat{y}_i(x) < L_i \\ \left(\dfrac{\hat{y}_i(x) - L_i}{T_i - L_i}\right)^s & \text{if } L_i \le \hat{y}_i(x) \le T_i \\ \left(\dfrac{\hat{y}_i(x) - U_i}{T_i - U_i}\right)^t & \text{if } T_i \le \hat{y}_i(x) \le U_i \\ 0 & \text{if } \hat{y}_i(x) > U_i \end{cases} \tag{11.28}$$

where the exponents s and t decide how strictly the target value is desired. When $s = t = 1$, the desirability function increases linearly towards T_i, the function is convex when $s < 1$, $t < 1$ and the function is concave for $s > 1$, $t > 1$.

If as an alternative a response is to be maximized, the individual desirability is instead defined as

$$d_i(\hat{y}_i) = \begin{cases} 0 & \text{if } \hat{y}_i(x) < L_i \\ \left(\dfrac{\hat{y}_i(x) - L_i}{T_i - L_i}\right)^s & \text{if } L_i \le \hat{y}_i(x) \le T_i \\ 1.0 & \text{if } \hat{y}_i(x) > T_i \end{cases} \tag{11.29}$$

where in this case T_i is considered as a large enough value for the response.

Finally, if we are interested to minimize a response, we could use

$$d_i(\hat{y}_i) = \begin{cases} 1.0 & \text{if } \hat{y}_i(x) < T_i \\ \left(\dfrac{\hat{y}_i(x) - U_i}{T_i - U_i}\right)^s & \text{if } T_i \le \hat{y}_i(x) \le U_i \\ 0 & \text{if } \hat{y}_i(x) > U_i \end{cases} \tag{11.30}$$

where T_i signifies a small enough value for the response.

Summary

- Properly designed experiment saves both our time and money. DOE method is used to plan the experiment before it started. Various experimental designs such as Box–Behnken, Central Composite etc. have been discussed in this chapter. The experimental data is utilized to find the model equation. Some statistical analysis is required for this purpose. The best-fit model is used for optimization using response surface methodology. Contours are also useful for finding the optimum point.

Review Questions

11.1 Why DOE is necessary for chemical process design?

11.2 What are the different stages used for DOE?

11.3 What is meant by replication? Why it is required?

11.4 Show that the Box–Behnken design of 3 factors is not a rotatable design.

11.5 Calculate the number of experiment required for B–B design with 4 factors.

11.7 When fractional factorial design is useful?

11.8 Show that a CCD is rotatable when $\alpha = \left(n_F\right)^{1/4}$ where n_F is the number of points used in the factorial portion of the design.

11.9 Show that using the relation $\alpha = \sqrt{k}$ results in a rotatable CCD for $k = 2$ and $k = 4$, but not for $k = 3$.

11.10. Consider the following response surface of two factors:

$$\hat{y}_i = 70 + 0.1x_1 + 0.3x_2 + 0.2x_1^2 + 0.1x_2^2 + x_1x_2$$

a. Find the coordinates of the stationary point.

b. What type of response function is this?

11.11 What do you mean by D-Optimal design?

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